The Role of Electron Microscopes in Nanoscience and Technology

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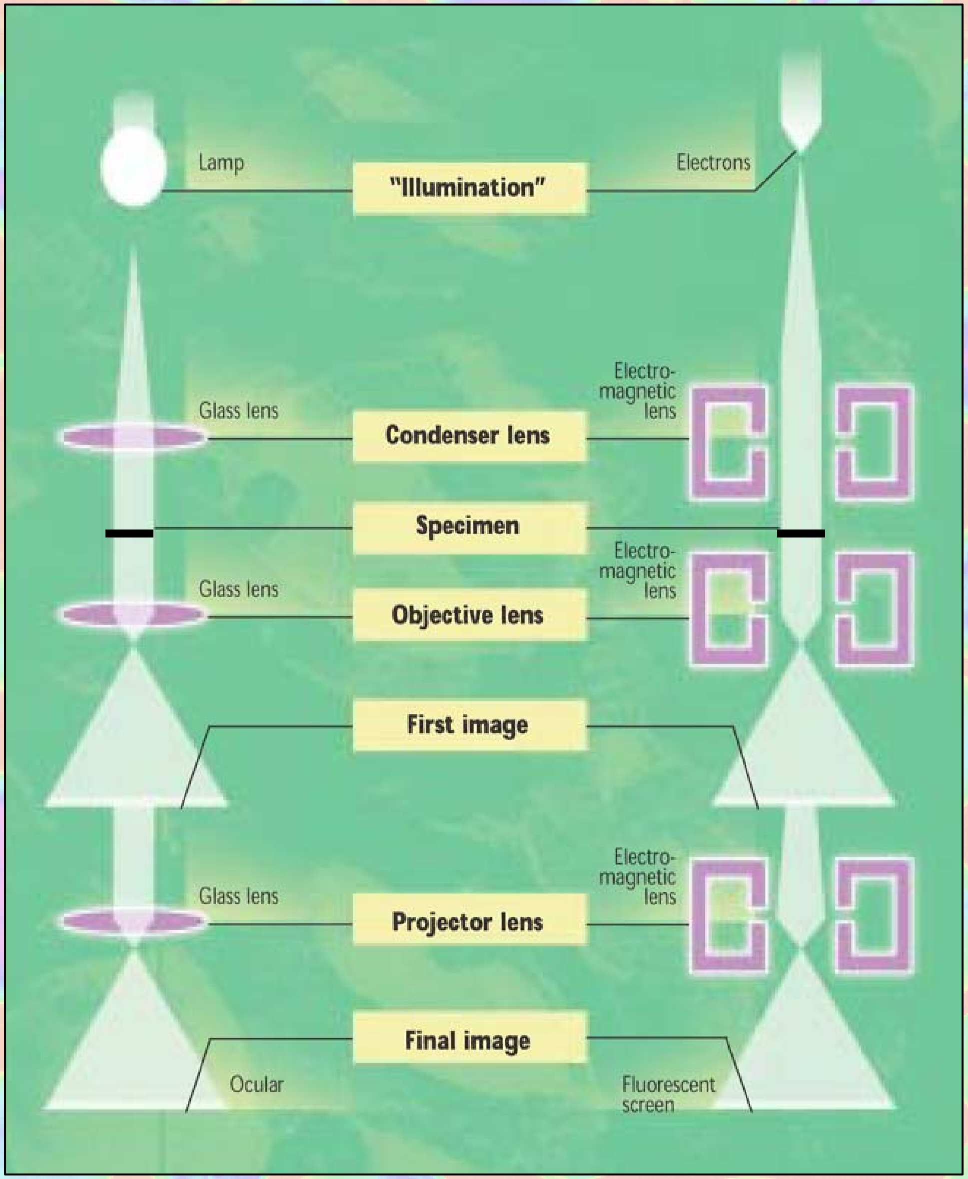
Electron Microscopes

- Transmission electron microscope (TEM)
- Scanning electron microscope (SEM)
- Scanning TEM (STEM)
- Focused ion-beam microscope (FIB)

This combination of instruments forms a comprehensive electron microscope laboratory

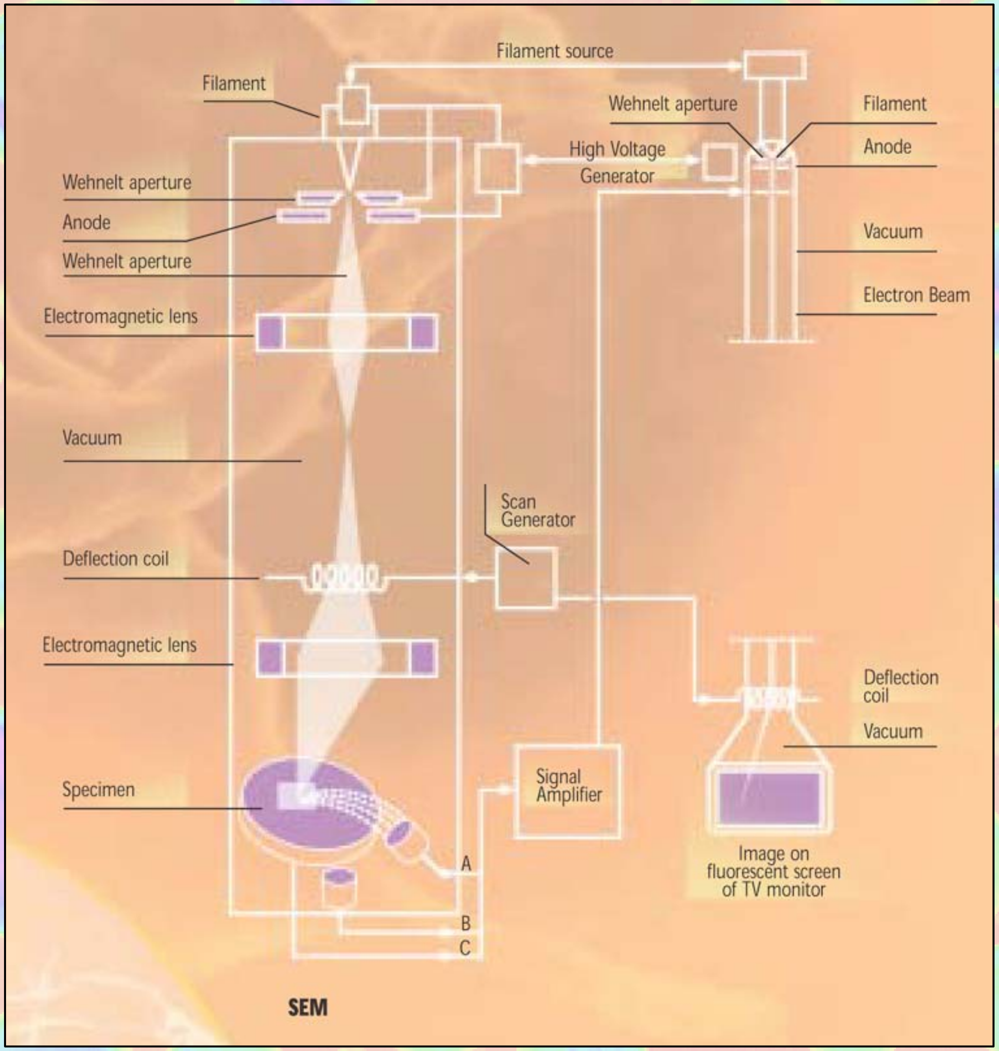
What is a TEM?



JEOL JSM-3100F

Point resolution 0.17 nm

What is an SEM?

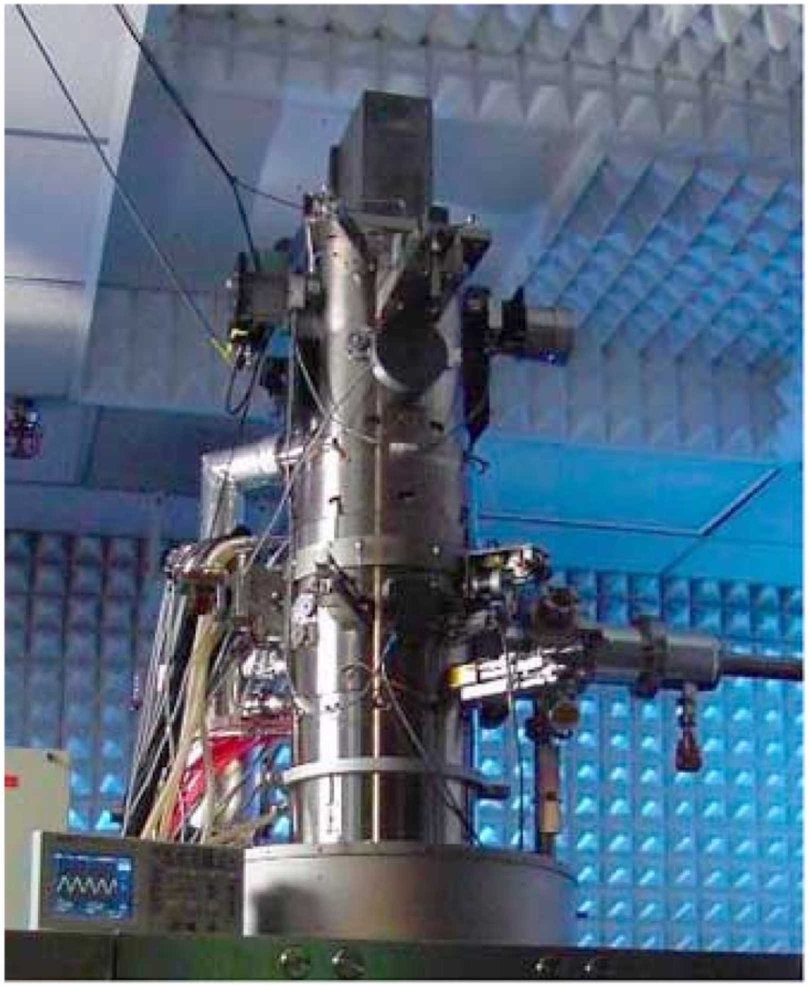


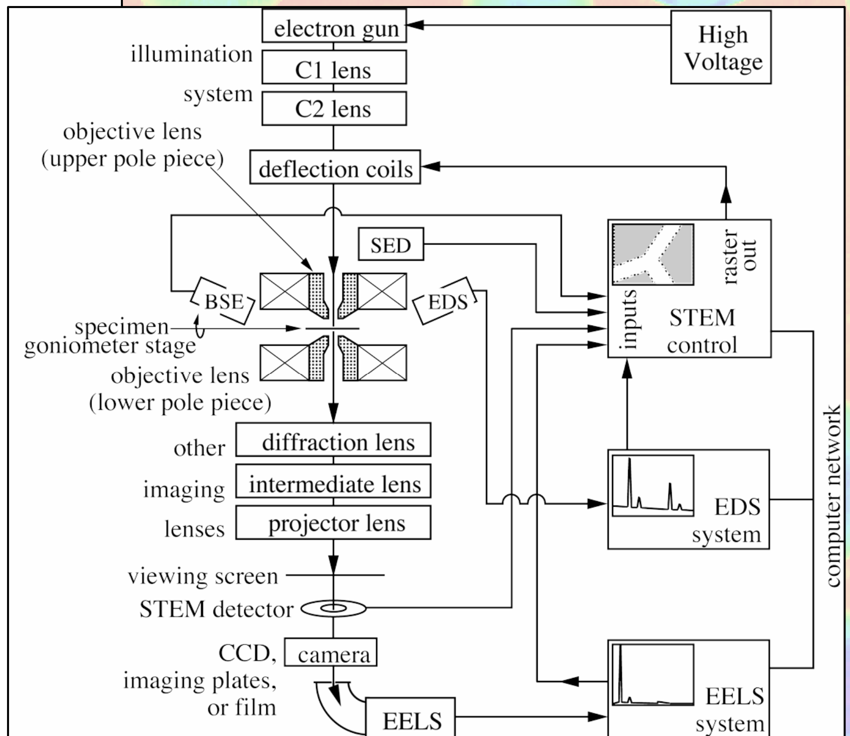
JEOL 7700F SEM

Point resolution 0.6 nm

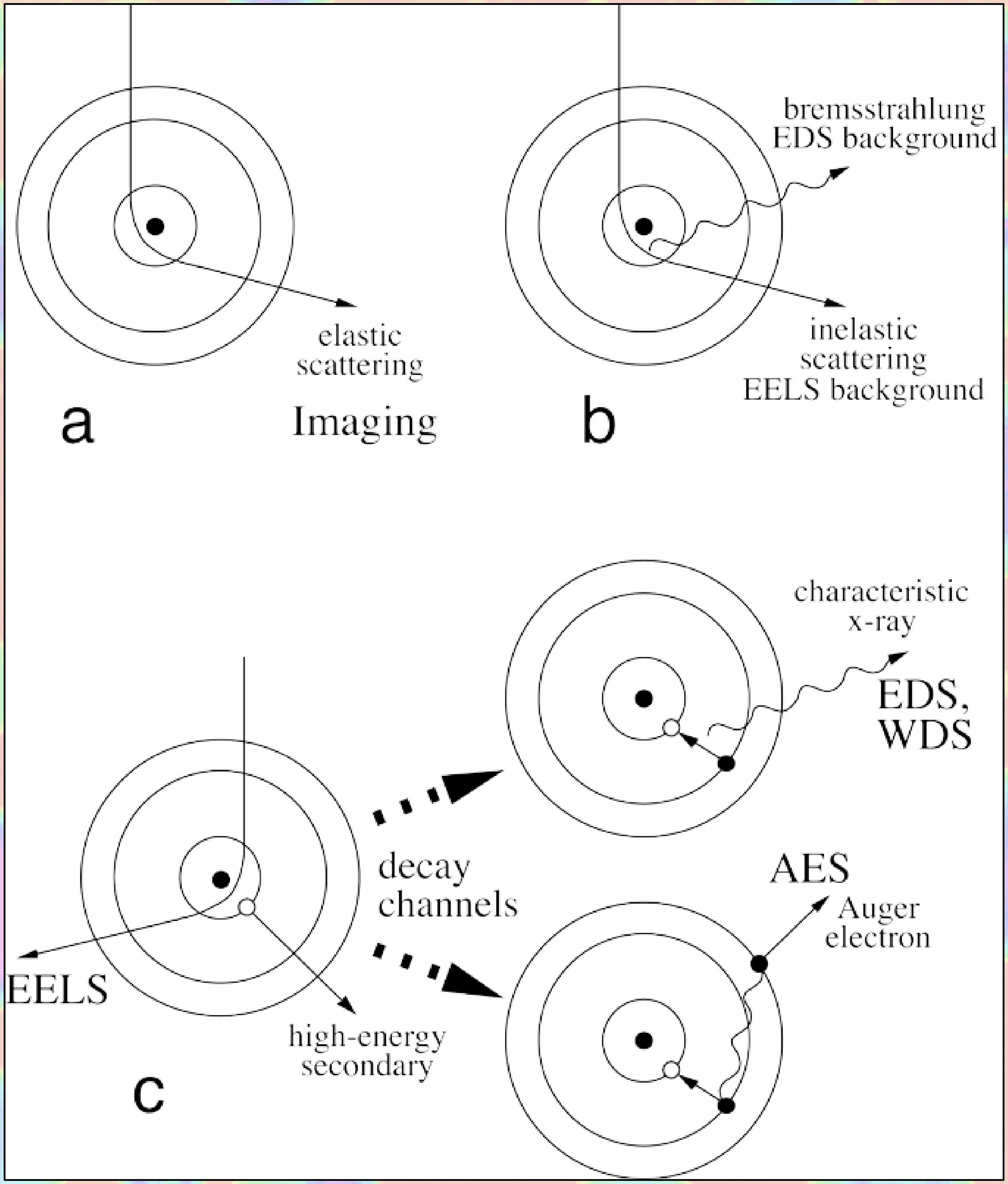
What is a STEM?

VG Microscopes HB603U STEM with Nion aberration corrector:





Point resolution ~ 0.06 nm



Spectroscopy in SEM and (S)TEM

EDS is better for high-Z elements; EELS is better for low-Z elements (e.g., B, C, O, N)

Resolution is approximately electron beam size in (S)TEM, limited to ~1 μm in SEM

What is an FIB?

FB-2100 FIB System

Product Number:FB-2100

Overview

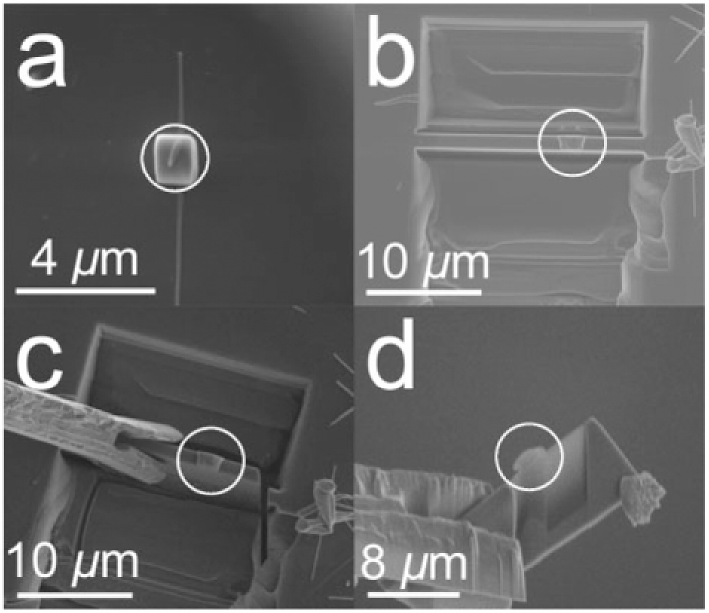


Figure 4. The FIB lift-out technique. The target area is circled in all four images. a) The target area is protected with a EBID-Pt capping layer. b) Trenches are cut on both sides of the target area. c) The micromanipulator is used to pick up the slab for further thinning. d) A side view of the slab shows that thinning has made the target area almost electron transparent.

- Accelerating Voltage 10kV - 40kV
- High Current Density ~ 30 nA
- In-Situ Lift Out Microsampling
- Built-in Scanning Ion Microscope (SIM) Imaging Capabilities with Resolution Less Than or Equal to 6nm

TEM/STEM

The addition of spherical aberration correctors and monochromators to the TEM/STEM improves spatial, chemical and bonding information (< 0.1 nm, < 0.15 eV)

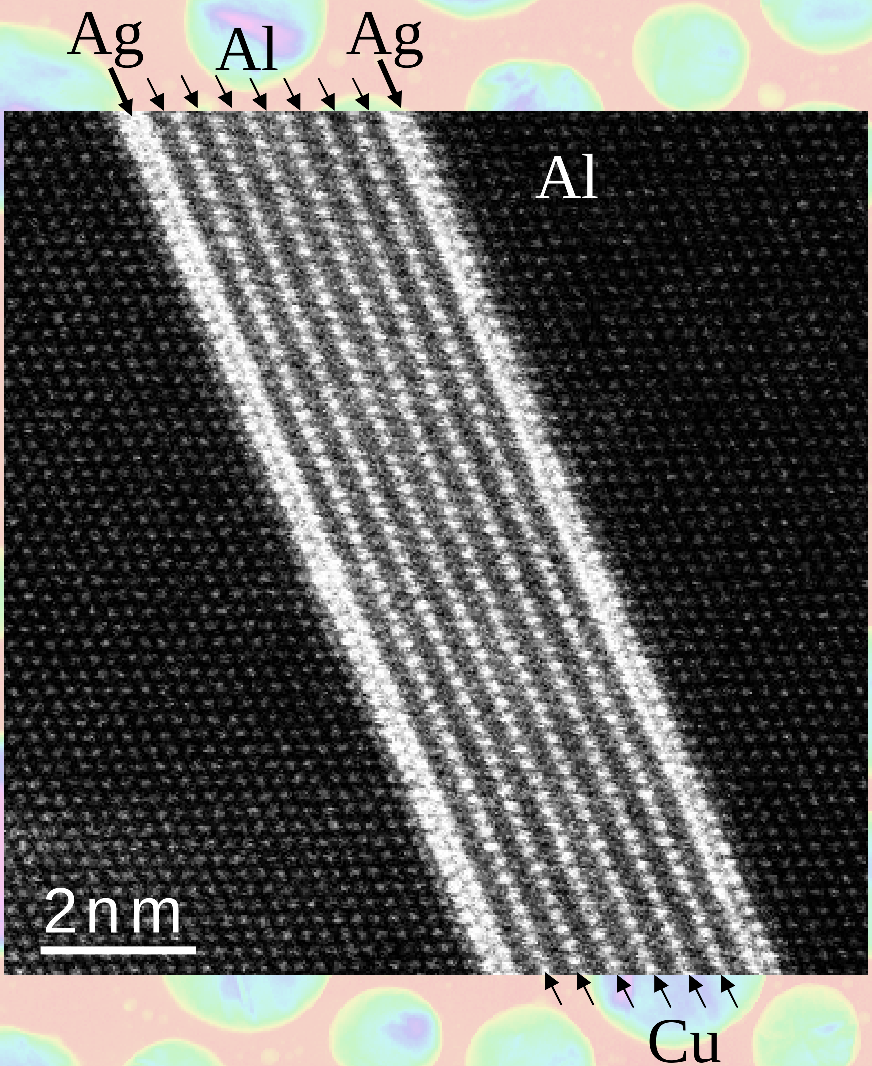
3-D tomography in TEM or STEM provides 3-D structural and chemical information

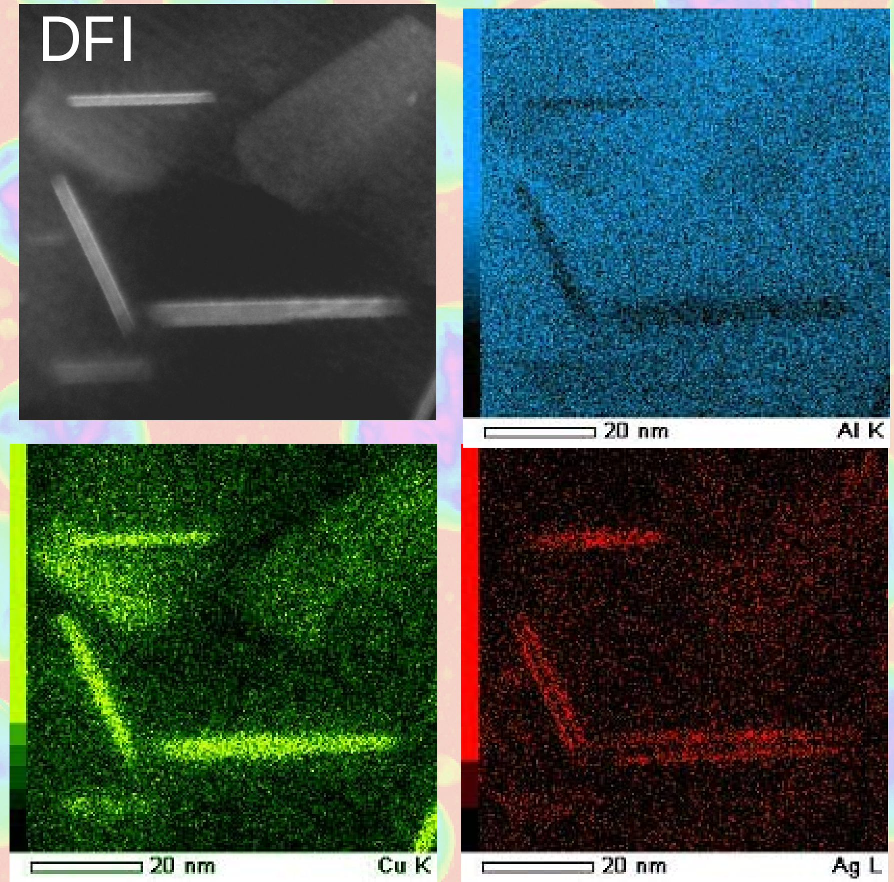
Use of the TEM as a nano-laboratory: ability to heat, cool, strain, indent, apply potential, laser light, magnetic field, a gaseous environment, often simultaneously

Use of TEM to determine bonding, physical and mechanical properties by electron energy-loss spectroscopy (EELS)

Laser-pulsed filaments for nanosecond time resolution

STEM EDXS mapping (with C_s corrector)

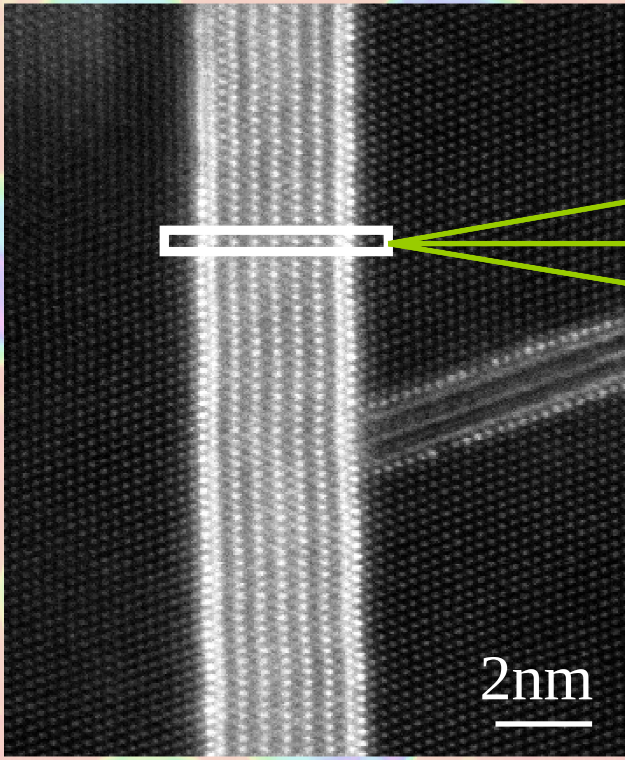


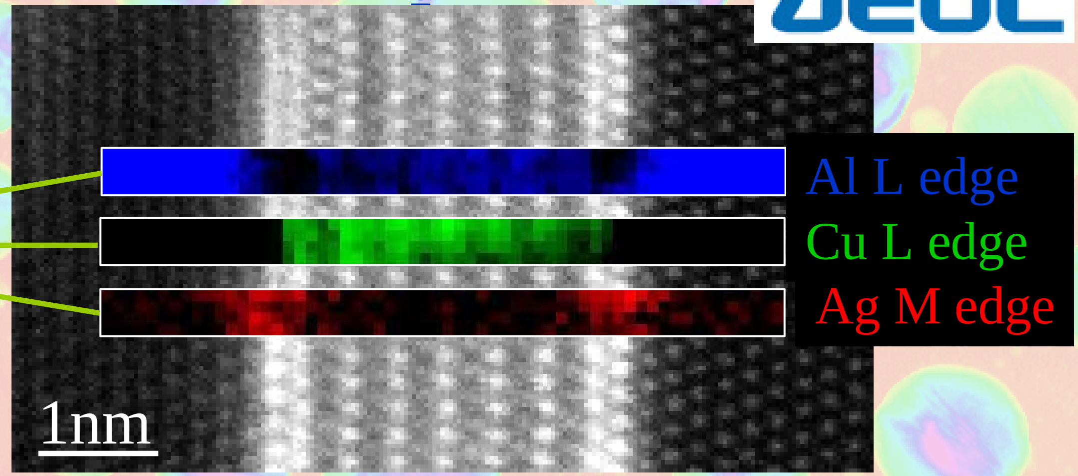


500pA□20min

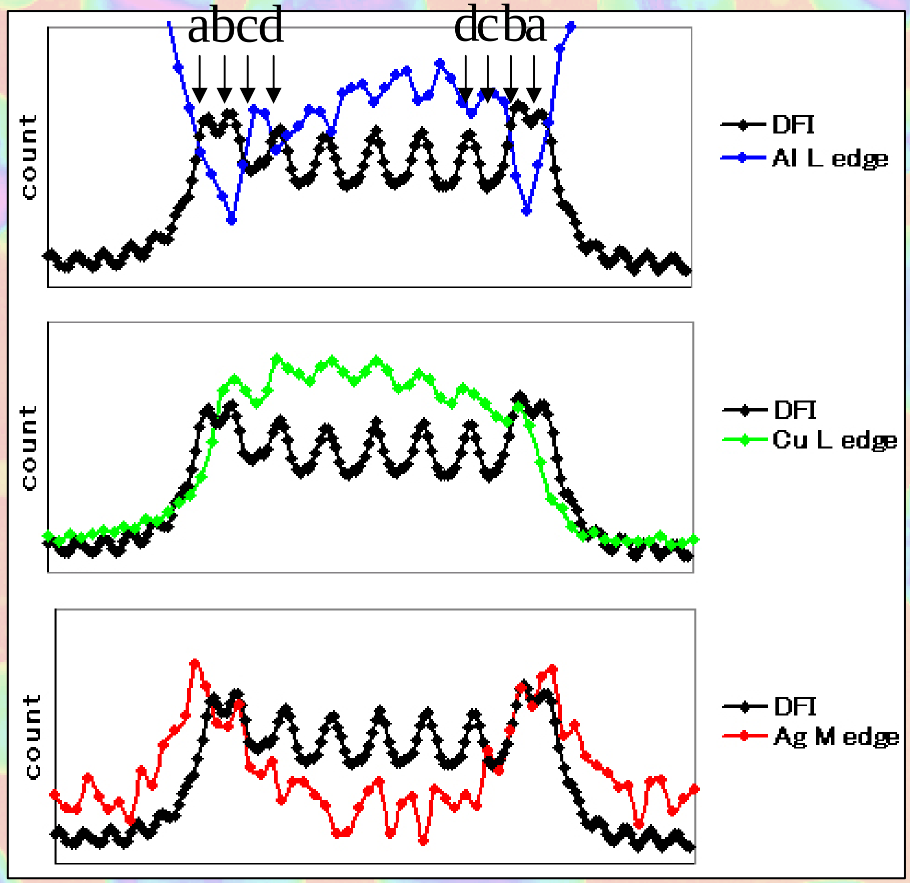
Specimen: courtesy of Prof. Hono, NIMS

STEM EELS atomic resolution mapping (with C_s corrector)

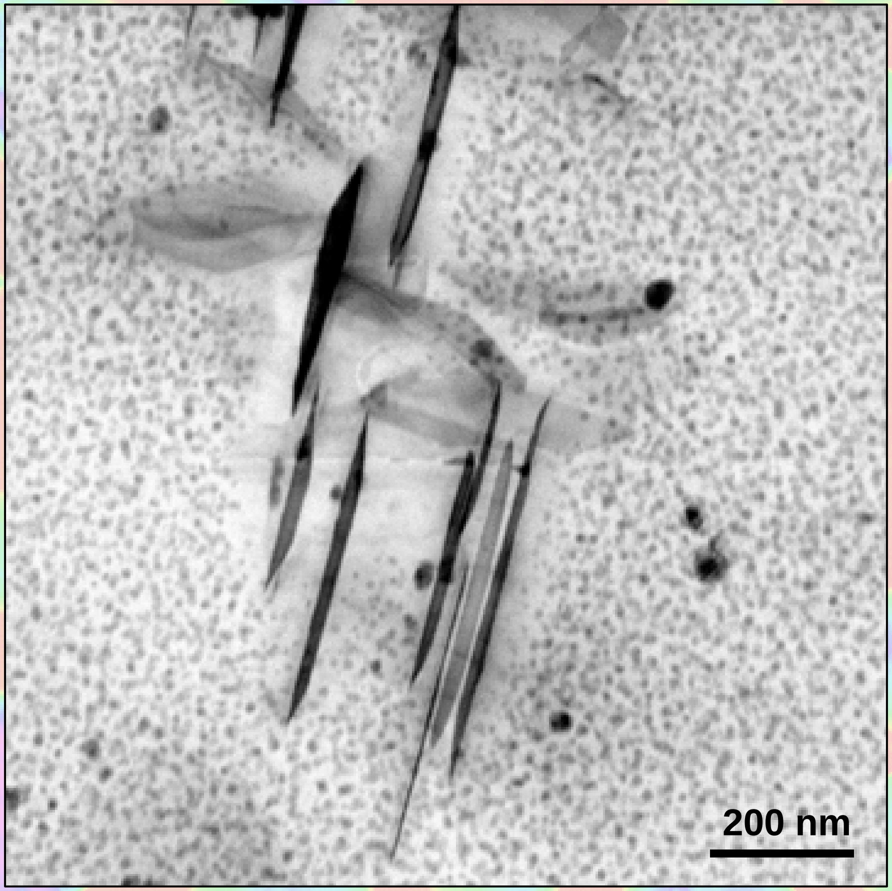


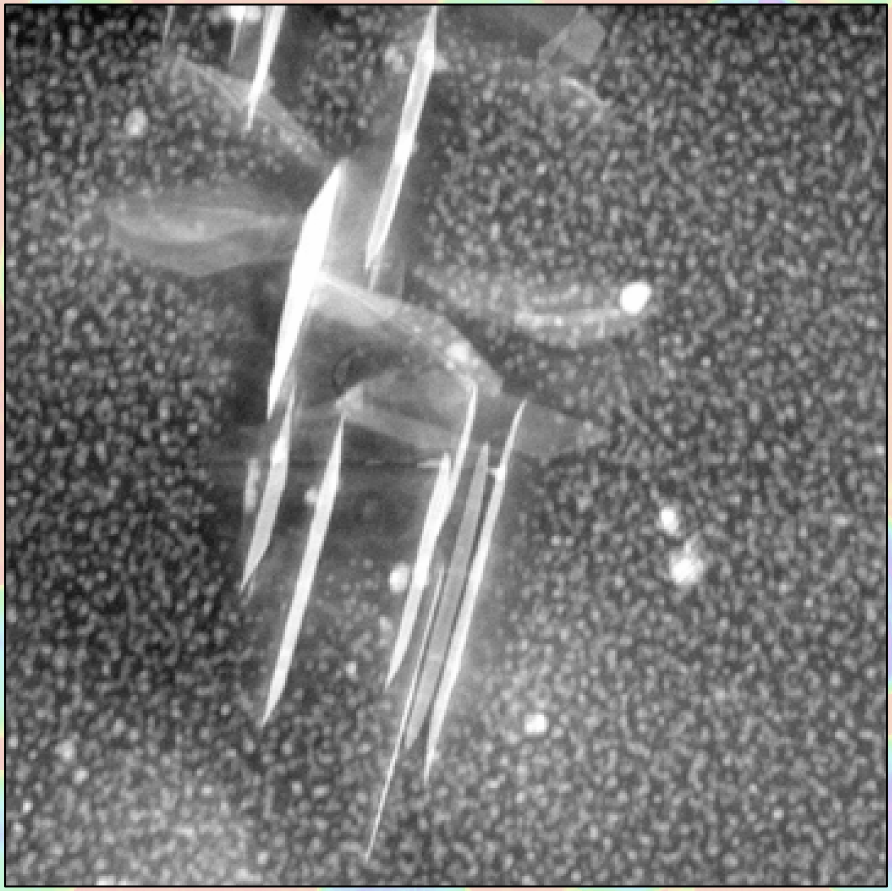


60pA□0.5s/pix (total 60x4 pix)



G.P. zones and γ' precipitates in Al-Ag alloy

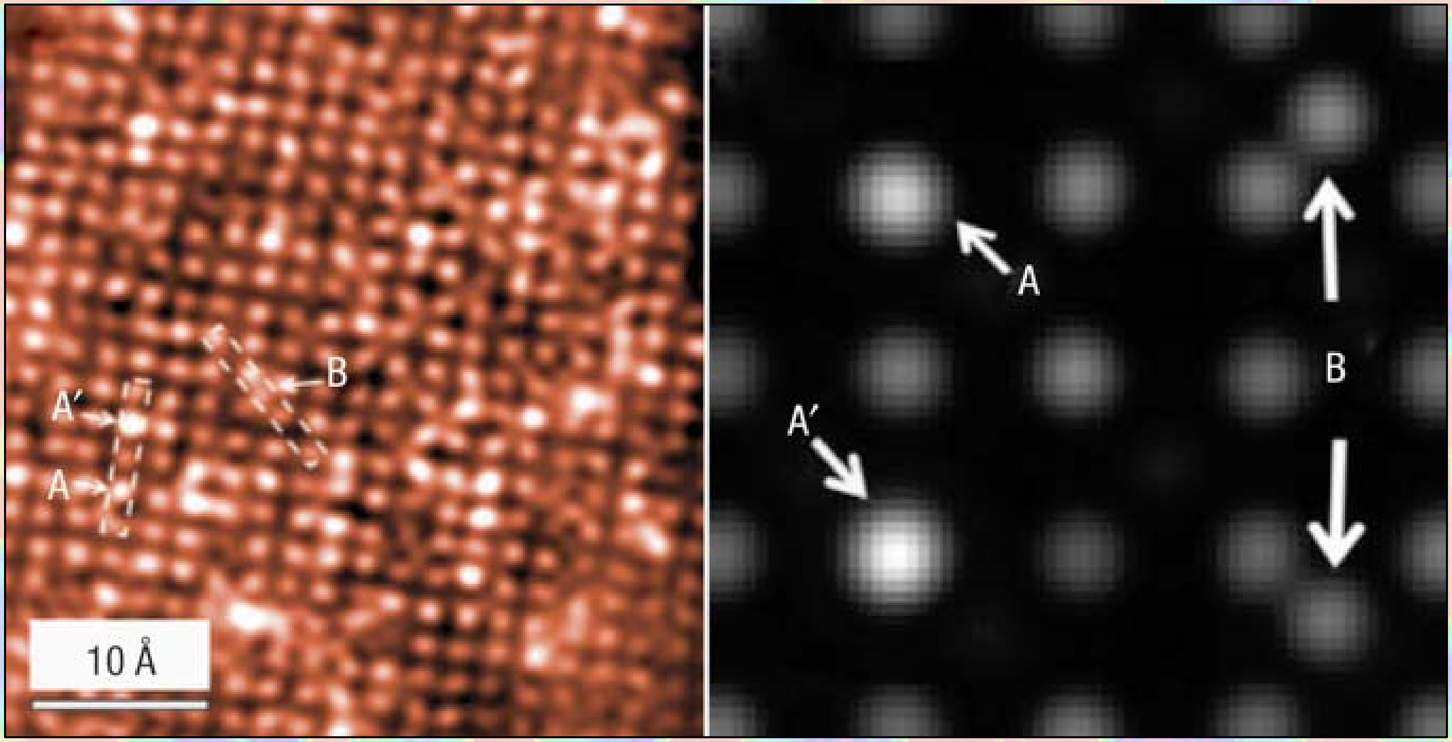


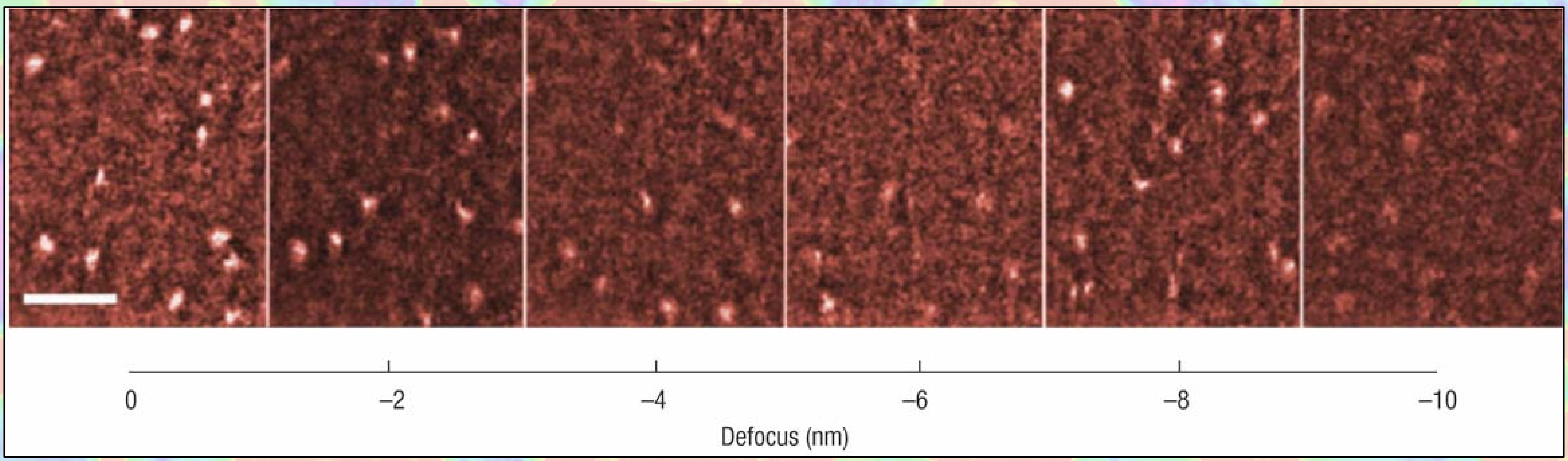


Bright-field TEM image **High-angle annular dark-field image**

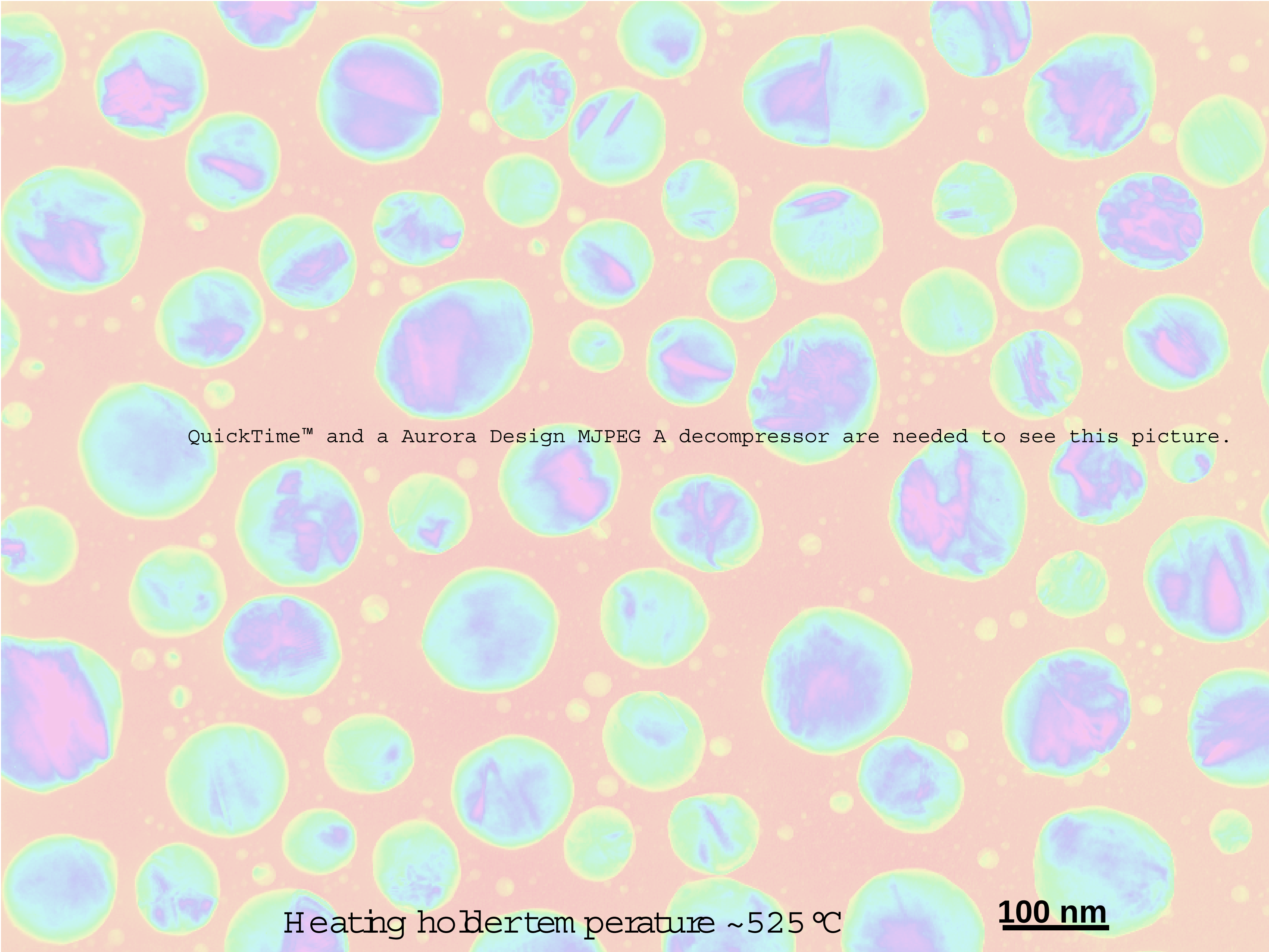
FEI Tokyo Demo - specimen courtesy of Dr. Kaneko, Kyushu Univ.

STEM 3D HAADF (FEI Tokyo Demo)





Wang et al., Nature Mater. Lett., 3 (2004) 143


QuickTime™ and a Aurora Design MJPEG A decompressor are needed to see this picture.
Heating holder temperature ~525 °C
100 nm

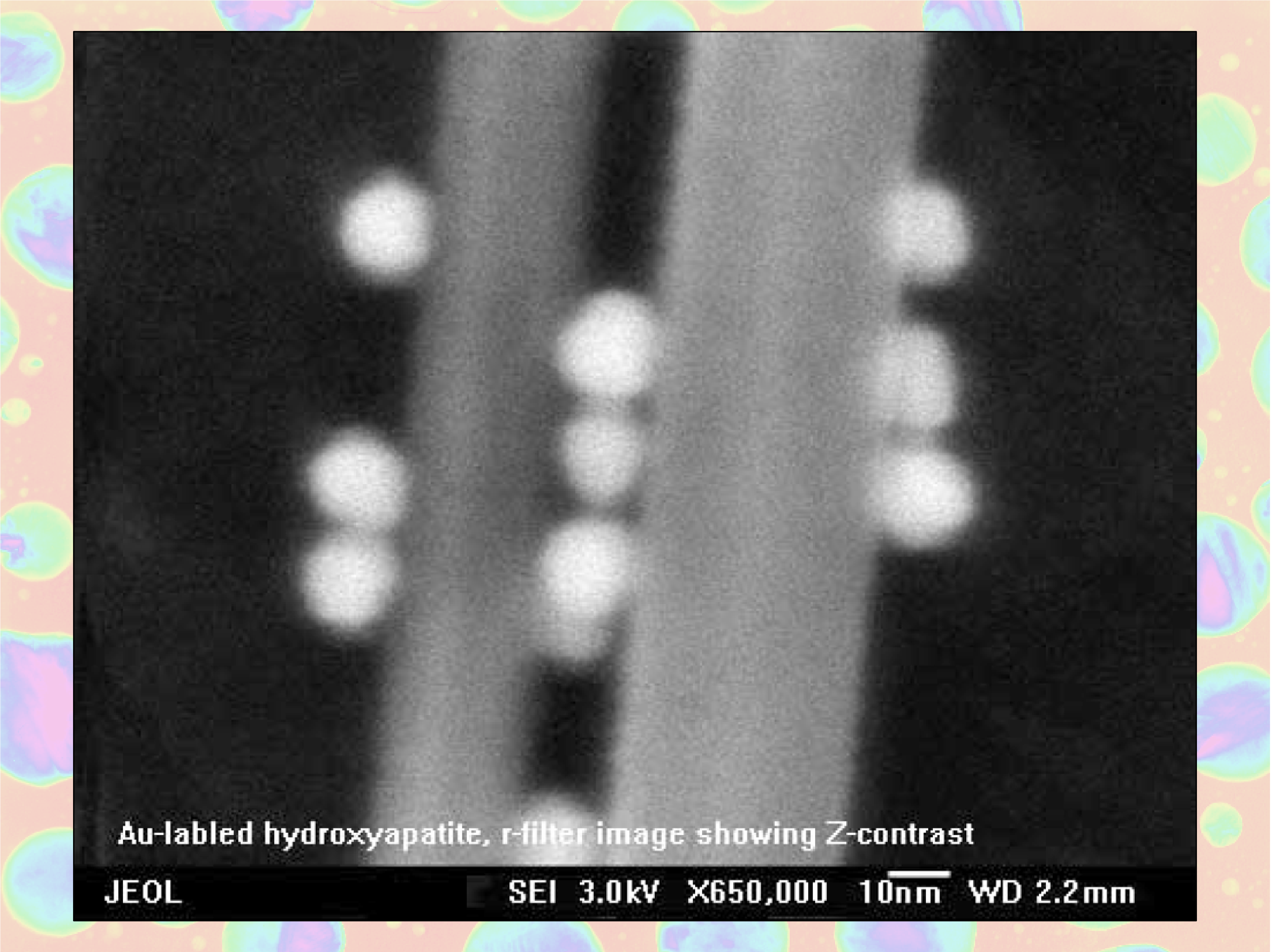
SEM

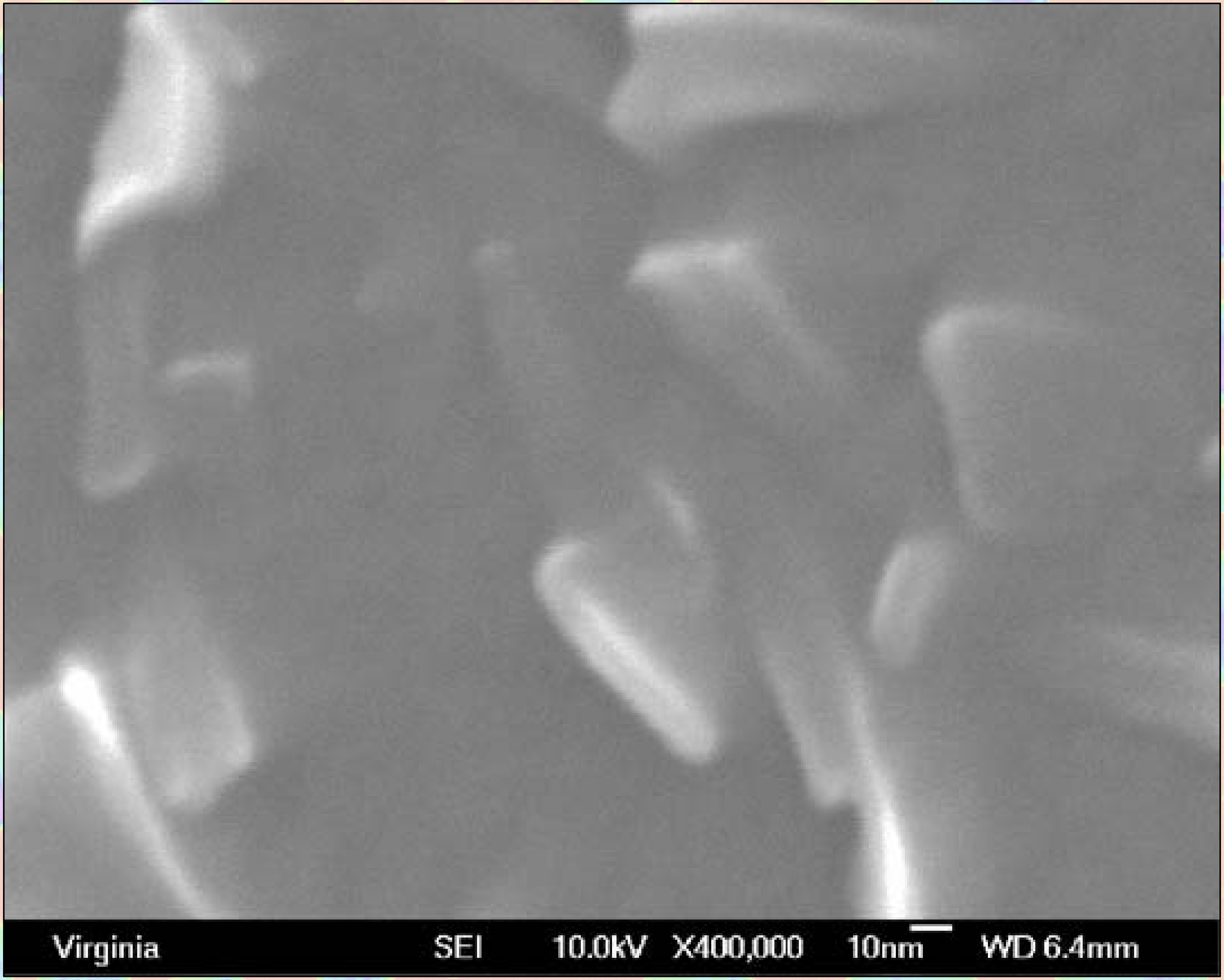
The current image resolution of field-emission gun SEM's is ~ 3 nm; this will continue to improve with the incorporation of C_s correctors and various lens/detector combinations (recently 0.5 nm, Hitachi)

Orientation imaging has become common and applied with great success to metal alloys; one of the challenges here is to improve the spatial resolution and sensitivity

SEM has essentially the same in-situ capabilities previously mentioned for TEM, plus wet cells, cathodoluminescence and more

Chemical analysis in the SEM is still limited by the interaction volume, although the use of thin specimens with special holders and lower accelerating voltages improve this (e.g., ~20-50 nm at 3 kV)


Au-labled hydroxyapatite, r-filter image showing Z-contrast
JEOL
SEI 3.0kV X650,000 10nm WD 2.2mm



C nanohorns - courtesy J. Fitz-Gerald, UVA

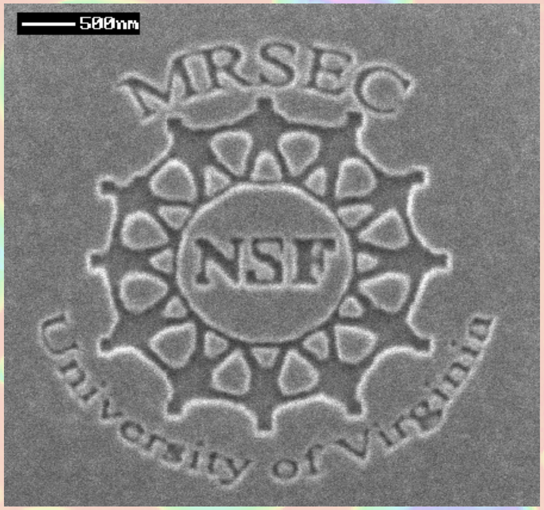
FIB

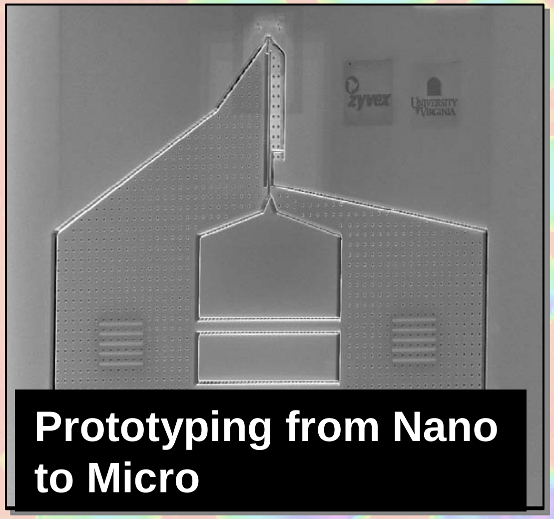
The FIB continues to be an important tool for TEM sample preparation, allowing thin foils to be fabricated from specific areas of bulk specimens (needle in a haystack problem) using the lift-out technique

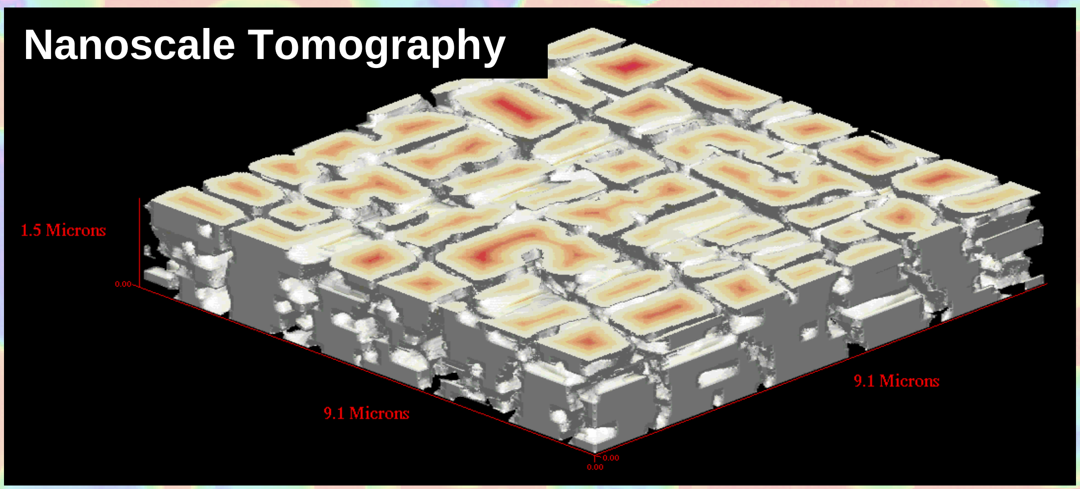
It allows one to section through metal alloys and form 3-D images or chemical maps (SIMS or EDXS) with excellent spatial resolution (~ 5 nm for imaging), and to view and analyze features below the surface; this has been enhanced by the advent of dual-beam FIB’s

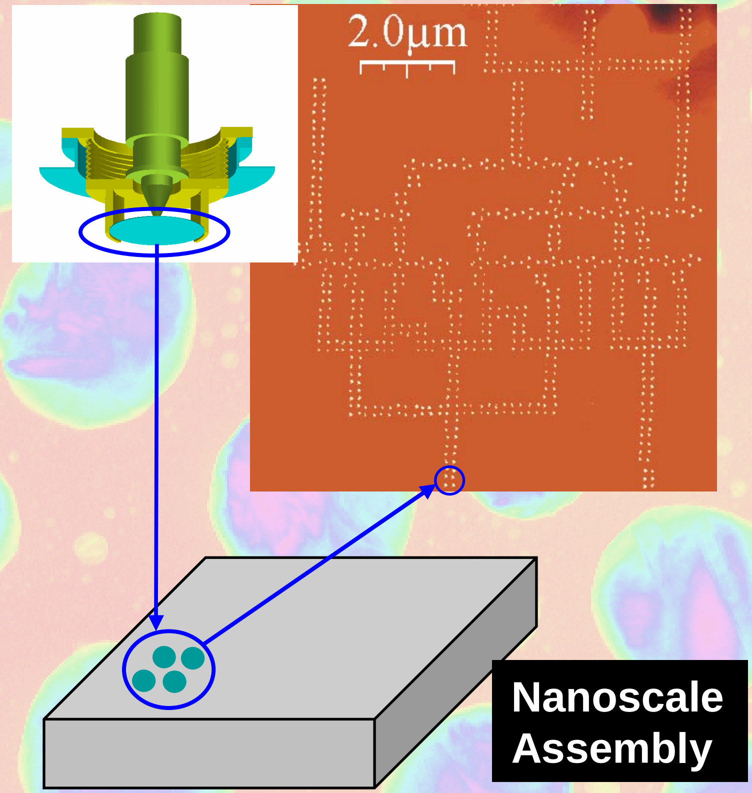
Surface damage can be an issue for metal specimens, limiting the specimen thickness for TEM samples, particularly for high-resolution TEM and EELS

The Focused Ion Beam – New Methods for 3D Imaging and Fabrication at the Nanoscale









Nanoscale Tomography
Source Development
Templated Asembly

Courtesy R. Hull, UVA

Nanoscale Materials Characterization Facility

SRA5

What is it?

It is a state-of-the-art facility dedicated t SRA6 microscopy, chemical and structural characterization of materials from atomic to microscopic levels.

- **Center has 3 TEM's, 2 SEM's, a FIB microscope, 3 X-ray XRD's, extensive specimen preparation, computation and analysis facilities.**
- **Some capabilities include:**
  - **Atomic imaging of materials with a resolution of 0.14 nm**
  - **Elemental analysis of regions as small as 0.5 nm in diameter**
  - **Dynamic experiments employing heating, cooling, straining, lasing**
  - **Orientational mapping and cathodoluminescence of materials** SRA7
  - **Electron-beam lithography of semiconductors/MEMS device fabrication**
  - **Controlled deposition and sputtering for micromachining**

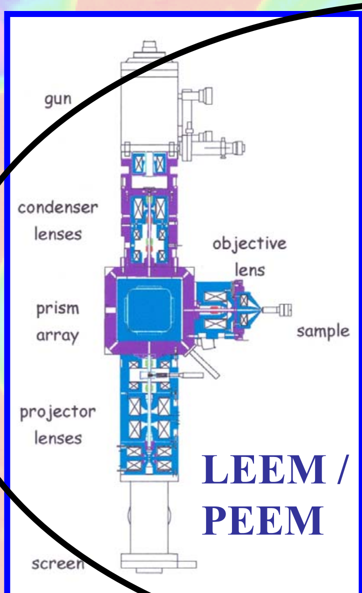
http://www.virginia.edu/ms/electronfacility.html

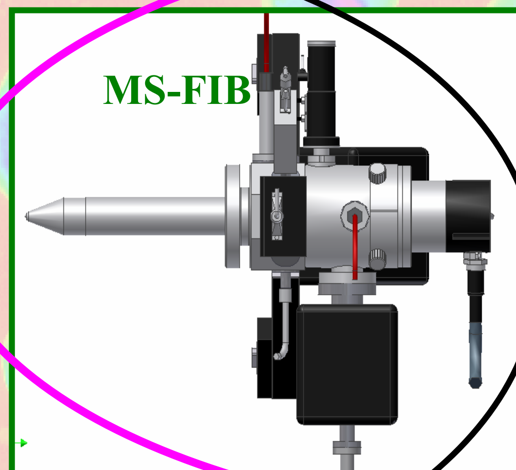
The Facility for Atomic-Scale Understanding of Nucleation and Assembly (FAUNA)

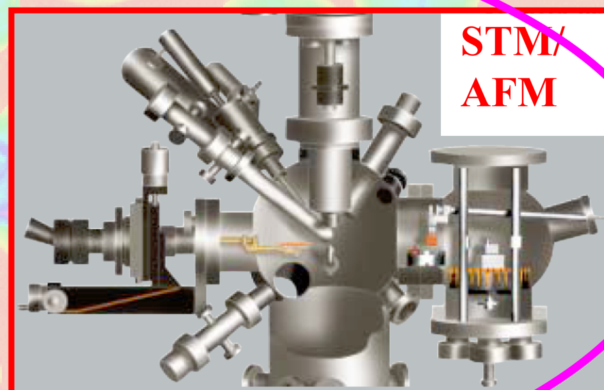
Funded, 2006

Here, in boxes

Here, operating



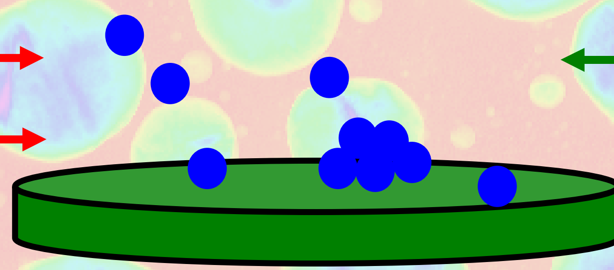




Vapor Delivery

Solid Source Deposition

Gas Source Deposition



Optical Beams

R. Hull

P. Reinke

QuickTime™ and a Cinepak decompressor are needed to see this picture.

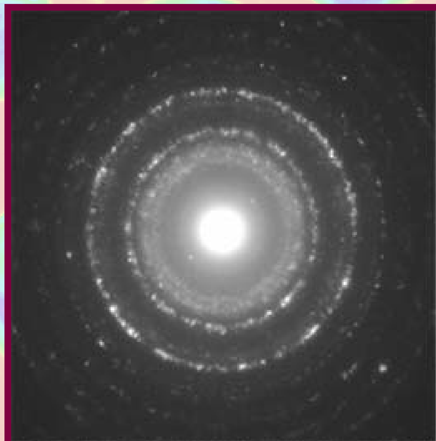
- **Real-time video of the dark-field (1/2, 0) from the FE LEEM P90 of O_2 etching Si (100) at 800°C**

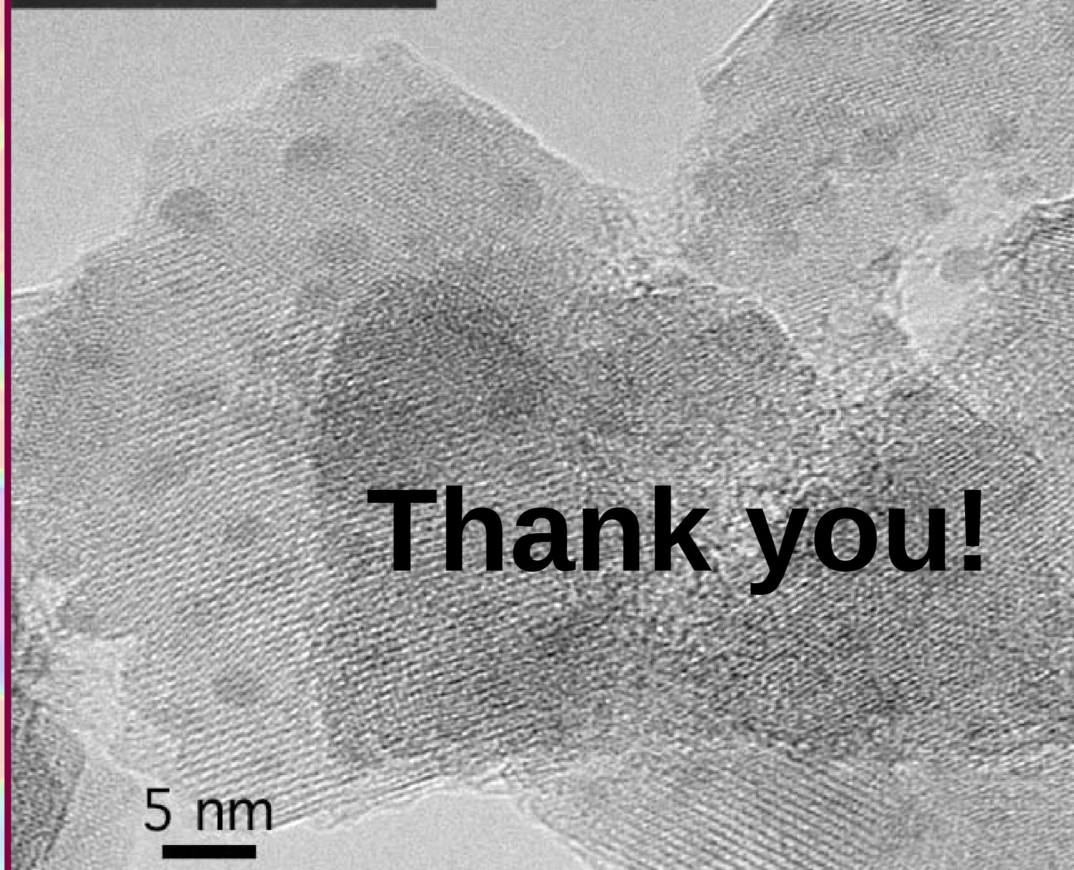
- **The partial pressure of O_2 is ˜ 1 x10-7 Torr and the field of view is 5.6 µm**

(R. Tromp, IBM)

Pd Deposited on γ-Al_2O_3 by PLD

R. Davis (CHE) and J. Fitz-Gerald (MSE)





Thank you!